

## Magnetic and transport properties of epitaxial Fe/V(001) superlattice films

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Single-crystalline Fe(15 ML)/V (1–12 ML) superlattices, epitaxially grown in the (001) direction, have been investigated using magnetization and electrical transport measurements. For all V thicknesses the system exhibits ferromagnetic properties. Results from magnetization and hysteresis measurements with the field applied in different directions confirm that the superlattice is well defined, the interface roughness is small, and the crystallinity of the samples is good. A clear influence of the V thickness, due to strain effects, on the in-plane magnetic anisotropy is found. Resistivity measurements show that the samples exhibit anisotropic magnetoresistive (AMR) properties with a strong dependence of the V thickness on the magnitude of the AMR. [S0163-1829(96)02326-0]

### I. INTRODUCTION

The magnetic and transport properties of thin magnetic films and multilayers have attracted considerable attention during the past years. The discoveries of the oscillating magnetic coupling<sup>1</sup> and giant magnetoresistance<sup>2</sup> in multilayers have led to intense research on multilayered systems consisting of 3d transition magnetic elements and spacer layers of 5d, 4d, or 3d nonmagnetic elements.<sup>3</sup> It has become clear that the material *combination* (not the individual materials) of the ferromagnetic and the nonmagnetic layers is of fundamental importance for the observed properties. Large magnetoresistance amplitudes have been found for many material combinations such as Fe/Cr, Co/Cu, Fe/Cu, and NiFe/Ag.<sup>4</sup>

Inspired by the high magnetoresistance values and the pronounced oscillating magnetic coupling in Fe/Cr,<sup>2,5</sup> theoretical and experimental investigations on the rather similar Fe/V system have been performed.<sup>6–9</sup> Vanadium, which is nonmagnetic in bulk form, apparently obtains an induced magnetic moment when forming a multilayered structure of Fe/V or when it is grown as an ultrathin film on single-crystalline Fe substrates. A study using spin-polarized electron-energy-loss spectroscopy<sup>7</sup> showed that 1 ML of V on Fe(001) orders magnetically in an antiparallel configuration with respect to Fe, while the surface layer in a 2-ML system couples ferromagnetically to the Fe substrate. This observation is in agreement with a self-consistent tight-binding calculation<sup>8</sup> and calculations performed by means of the interface Green's function technique<sup>9</sup> on V overlayers on bcc Fe(001). The theoretical work in Ref. 8 also covers calculations on a Fe/V(001) superlattice, where it was found that V orders antiferromagnetically within each layer. An extensive calculation, using a first-principle Green's-function technique on a bcc Fe/V/Fe(001) trilayer,<sup>10</sup> confirms that V obtains a magnetic moment, but a different configuration of the V moments was inferred. In the case where the Fe layers couple ferromagnetically all V moments are aligned antiparallel with respect to the Fe layer. When the Fe layers possess antiferromagnetic coupling, the V moments change direction

in the middle of the V layer so that an antiparallel alignment between the Fe and the nearest V atomic layers is obtained. The difference in energy between the ferromagnetic and antiferromagnetic configurations of the Fe layers determines the sign of the coupling energy, which oscillates with an increased number of atomic layers of V.

To our knowledge, no clear experimental evidence has been reported confirming the existence of oscillatory magnetic coupling in Fe/V multilayers. Results from (110) textured bcc Fe/V multilayers<sup>3</sup> have indicated a very weak coupling, whereas another experimental study<sup>11</sup> did not find any oscillatory coupling, but only magnetoresistive properties, which were assigned to hysteresis effects. In a study using neutron-scattering experiments on epitaxial Fe/V(001) multilayers, no antiferromagnetic exchange coupling was found for a spacer thickness of 9 Å.<sup>12</sup>

In the present study we investigate single-crystalline superlattices of Fe/V(001) with the nominal structure MgO(001)/[Fe(15 ML)/V( $N_V$  ML)]<sub>20</sub>/Pd(30 Å), where  $1 < N_V < 12$  ML. Superconducting quantum interference device (SQUID) magnetometry shows that the system exhibits ferromagnetic properties for all  $N_V$ ; i.e., no sign of antiferromagnetic coupling was observed. Magnetoresistance measurements indicate that all samples exhibit anisotropic magnetoresistance (AMR). The magnitude of the AMR decreases monotonically with increasing number of atomic V layers.

### II. EXPERIMENT

The Fe/V superlattices were produced in a three-source ultrahigh vacuum (UHV) based sputtering system with an *in situ* reflection high-energy electron diffraction and residual gas analyzer. The substrates, polished single-crystalline MgO(001) wafers ( $10 \times 5 \times 0.5$  mm<sup>3</sup>), were all from the same batch. The substrates were ultrasonically precleaned using isopropanol and ethanol, before loading into the deposition system and outgassing in UHV at 700 °C for 10 min. The base pressure of the system was  $10^{-9}$  Torr during annealing. During sputtering, the Ar (99.9999%) pressure was

kept at  $5 \times 10^{-3}$  Torr, giving a typical deposition rate of Fe and V of 0.5 Å/s. The deposition rate was monitored by quartz crystal microbalances (QCM's) placed in front of each magnetron. During deposition, the substrate was rotated ( $\approx 100$  rpm) and kept at 330 °C, which is the optimal growth temperature for this system.<sup>13</sup> To avoid mixing of Fe and Pd, the Fe/V superlattice was always finished with a V layer before deposition of the Pd capping layer.

The structural quality of the samples, the individual layer thicknesses of Fe and V, the average out-of-plane lattice parameter  $\langle c \rangle$ , and the modulation, wavelength were determined using conventional  $\theta$ - $2\theta$  x-ray diffraction (XRD) (Cu  $K\alpha$  radiation). The measurements were carried out in a low-angle region ( $1$ – $12^\circ$  in  $2\theta$ ) as well as in a high-angle region ( $50^\circ$ – $80^\circ$  in  $2\theta$ ) around the Fe/V(002) Bragg peak. A Siemens powder diffractometer equipped with a Eulerian cradle and a secondary monochromator was used for the XRD measurements. The angular resolution for the low- and high-angle scans was  $0.005^\circ$  and  $0.01^\circ$ , respectively. From the QCM measurements and simulations of the low-angle reflectivity data the individual Fe and V thicknesses could be determined. In a previous extensive investigation concerning the growth of Fe/V(001) superlattices,<sup>13</sup> a Frank–van de Merwe growth of Fe/V superlattices with good crystal quality and an average interfacial roughness of typically  $\pm 1$  Å was obtained in the temperature region 310 °C–330 °C.

For the magnetization and the resistivity measurements the samples were cleaved into square pieces, approximate size  $3 \times 3$  mm<sup>2</sup>, with the [100] and [010] directions of the MgO substrate parallel to the edges. According to the diffraction study,<sup>13</sup> the epitaxial relationships between the deposited film and the substrate are Fe/V(001)||MgO(001) and Fe/V[110]||MgO[100]; the [110] directions of the Fe/V superlattice is therefore parallel to the edges of the substrate. Magnetization measurements were performed in a Quantum Design 5.5-T SQUID magnetometer. In these measurements the magnetic field was applied in the film plane to avoid demagnetization effects. The resistivity measurements were carried out in a LakeShore 7225 series Susceptometer/Magnetometer system. The resistivity probe is configured in a four lead geometry. The measurements were performed in the dc mode at  $I = 1$  mA, giving a current density of  $\approx 10^7$  A/m<sup>2</sup> in the samples. To avoid influences of thermal electric power, the resistivity was determined as the average value of the recorded data in the forward and reverse current directions.

### III. RESULTS

#### A. Magnetic properties

Typical results from the magnetization measurements are shown in Fig. 1, where the reduced magnetization is plotted vs applied field at 10 K for the Fe/V (7-ML) sample. The different curves in Fig. 1(a) corresponds to fields applied in the [100] and [110] directions. In Fig. 1(b) the low-field part of a full magnetization loop in the [110] direction is plotted. In the easy [100] direction the magnetization immediately saturates at very low fields, whereas in the hard [110] direction the magnetization reaches the value  $(1/\sqrt{2})M_{\text{sat}}$  at low fields and saturates at an applied field of approximately 53 kA/m. The full magnetization loop shows that the magneti-

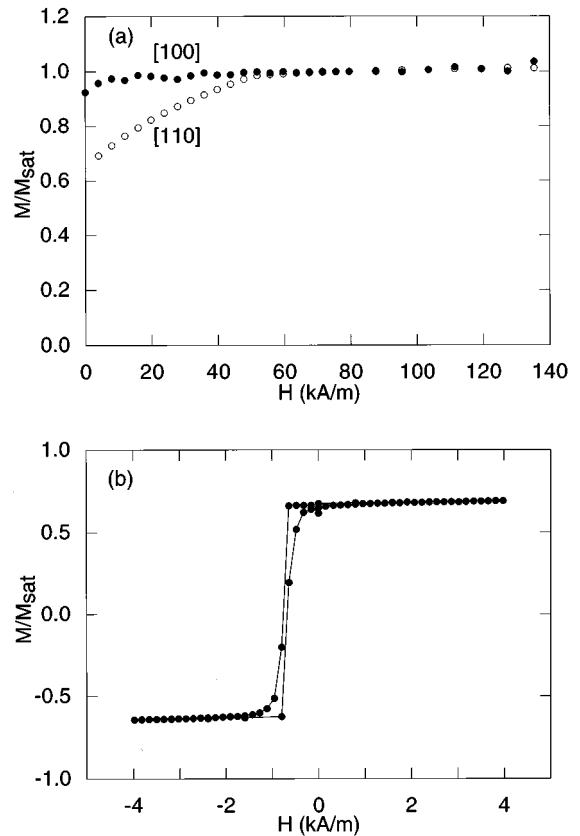


FIG. 1. (a) Reduced magnetization  $M/M_{\text{sat}}$  vs applied magnetic field in the [100] and [110] directions and (b) a full magnetization loop in the [110] direction for a single-crystalline Fe(15 ML)/V(7 ML) superlattice. The field is applied in the (001) plane of the film at  $T = 10$  K. The shift of the magnetization loop along the magnetic field axis in (b) is due to a remnant magnetic field in the superconducting solenoid.

zation reversal occurs in a very narrow field range, implying weak domain wall pinning effects. The typical ferromagnetic properties presented in Fig. 1 are representative for all samples with V thicknesses in the range  $1 < N_V < 12$  ML. No influence of the V thickness on the magnetic properties, which may indicate an antiferromagnetic coupling between the Fe layers, has been observed. The resemblance to single-crystalline bulk iron, regarding the orientation dependence of the magnetization<sup>14</sup> and the absence of hysteresis effects, confirm the epitaxial structure and the small interfacial roughness found from the structural characterization experiments.<sup>13</sup>

The saturation magnetization value was obtained using the volume of Fe and the measured magnetic moment in the film plane at 10 K. The total thickness of Fe in a sample was determined from the diffraction data. In Fig. 2 the saturated magnetization values are plotted for different vanadium thicknesses. The calculated values are close to the saturation magnetization of bulk Fe (2.2 T). The scatter in the data may be largely attributed to the uncertainty in the determination of the volume of Fe in the films. To estimate the contribution from a possible magnetic moment on vanadium to the total moment of a film, results from theoretical calculations have been used. According to these calculations,<sup>10</sup> only the vanadium atomic layer at the Fe/V interface has a significant

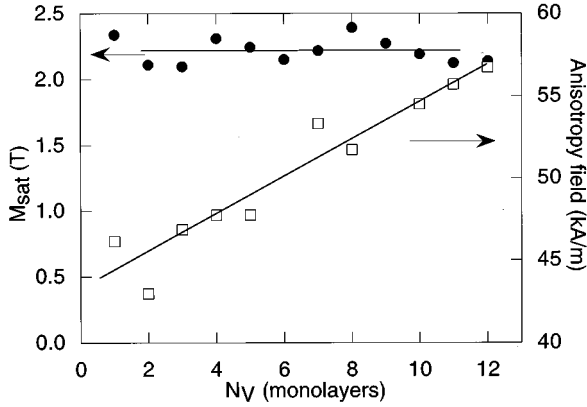


FIG. 2. Saturation magnetization (circles) and anisotropy field (squares) in the [110] direction as a function of the number of vanadium layers  $N_V$ . The data are extracted at 10 K. The horizontal line indicates the bulk magnetic moment for Fe; the sloping line is a guide to the eye.

moment ( $0.37\mu_B$ ). Using this value, the contribution from the V layers to the total magnetic moment of the films is at most 1–2 % and within the experimental error. In measurements performed in the direction perpendicular to the film plane, the magnetization increases linearly with applied field and saturates at a field of approximately  $H_{\text{sat}} \approx 1750 \pm 80$  kA/m. Due to the demagnetization effects the magnetization follows  $M = (1/N)H$ , where  $N$  is the demagnetization factor. Using  $N = 1$  in the perpendicular direction, a saturation magnetization of  $M_s = \mu_0 H_{\text{sat}} \approx 2.2 \pm 0.1$  T is obtained. The agreement with the directly measured magnetic moment implies that any imposed dipolar perpendicular anisotropy in the films due to interface roughness is negligible.<sup>15</sup>

The higher anisotropy field [as seen in Fig. 1(a)] for the Fe/V system, compared to the value for bulk iron, is due to internal strains in the superlattice produced by differences in lattice parameters between Fe ( $a = 2.87$  Å), V ( $a = 3.03$  Å), and the substrate ( $a/\sqrt{2} = 2.97$  Å). To illustrate the impact of the strain, the anisotropy field (Fig. 2) and the average out of plane lattice parameter  $\langle c \rangle$  (Fig. 3) are plotted as a function of the number of V atomic layers. The average lattice param-

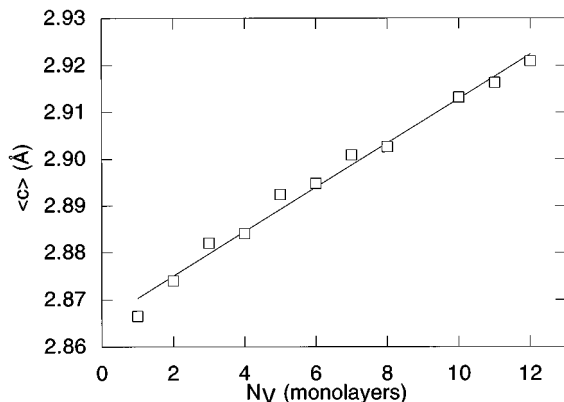


FIG. 3. Average out-of-plane lattice parameter  $\langle c \rangle$  as a function of the number of vanadium layers  $N_V$ .

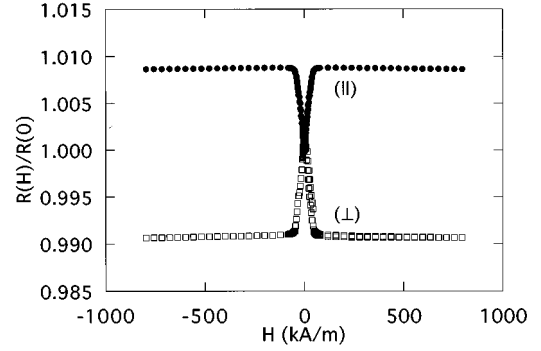


FIG. 4. Reduced electrical resistance  $R(H)/R(0)$  vs applied magnetic field  $H$  for the Fe(15 ML)/V(3 ML) superlattice. The magnetic field is applied in the film plane in the parallel (||) and perpendicular ( $\perp$ ) directions with respect to the current. The current is fed in the [110] direction.

eter, extracted from the location of the (002) Fe/V Bragg peak, is defined as the weighted average value of the out-of-plane lattice parameters  $a_{\perp}^{\text{Fe}}$  and  $a_{\perp}^{\text{V}}$  for Fe and V, respectively, according to

$$\langle c \rangle = \frac{N_{\text{Fe}} a_{\perp}^{\text{Fe}} + N_{\text{V}} a_{\perp}^{\text{V}}}{N_{\text{Fe}} + N_{\text{V}}}. \quad (1)$$

From the diffraction experiments it is found that for a given  $N_V$ , the out-of-plane lattice parameters for Fe ( $a_{\perp}^{\text{Fe}}$ ) and V ( $a_{\perp}^{\text{V}}$ ) are compressed and expanded relative to respective bulk values.<sup>13</sup> Due to the Poisson response, opposite changes of the in-plane lattice parameters occur: the in-plane lattice parameter for Fe,  $a_{\parallel}^{\text{Fe}}$ , is expanded, whereas  $a_{\parallel}^{\text{V}}$  is compressed. When the V thickness is increased the diffraction data show that the *average* out-of-plane lattice parameter increases [due to the influence of  $N_V$  on  $\langle c \rangle$  according to Eq. (1)] and that the out-of-plane lattice parameters for both Fe and V,  $a_{\perp}^{\text{Fe}}$  and  $a_{\perp}^{\text{V}}$ , successively *decrease*. This implies that a successive expansion of the in-plane lattice parameters occurs. Due to magnetoelastic effects, the expansion of the  $a_{\parallel}^{\text{Fe}}$  influences the in-plane magnetic anisotropy of iron. This effect is reflected as a dependence of the anisotropy field on the number of V atomic layers, as seen in Fig. 2.

## B. Transport properties

In Fig. 4 the magnetoresistance  $R(H)/R(0)$  for the Fe/V (3-ML) sample is plotted vs applied field in the [110] direction. The different curves correspond to feeding the current in the parallel ( $H \parallel I$ ) and in the perpendicular ( $H \perp I$ ) orientation with respect to the magnetic field. As can be seen in the figure, the behavior of the magnetoresistance is strongly dependent on the orientation of the magnetization with respect to the electric current. The same general behavior, as exemplified in Fig. 4, was found for all the samples independent of the number of atomic V layers. This is characteristic for the AMR, which yields different behavior of the resistance vs applied field depending on whether the current and the magnetization directions are parallel or perpendicular to each other. The AMR, which originates from the spin-orbit coupling, is a well-known effect in bulk ferromagnetic tran-

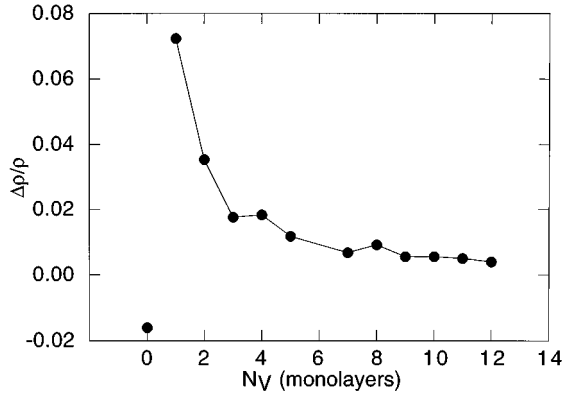


FIG. 5. Anisotropic magnetoresistance  $\Delta\rho/\rho$ , defined according to Eq. (4), vs the number of vanadium layers  $N_V$ .  $T = 10$  K.

sition metals and is a phenomenon fundamentally different from the giant magnetoresistance effect found in many multilayered systems. Since no giant magnetoresistance effect is observed in the present Fe/V multilayer system, it can be concluded that the magnetoresistance effect (AMR) arises from the magnetic properties of the individual Fe layers.

The spontaneous anisotropic magnetoresistance is defined as<sup>16</sup>

$$\Delta\rho/\rho = (\rho_{\parallel} - \rho_{\perp})/\rho_{\text{av}}, \quad (2)$$

where  $\rho_{\parallel}$  and  $\rho_{\perp}$  are the resistivities in the parallel and perpendicular orientations, respectively, obtained by extrapolating the high-field resistances to zero magnetic induction  $B = 0$ . For a three-dimensional system the average resistivity is defined as  $\rho_{\text{av}} = \frac{1}{3}\rho_{\parallel} + \frac{2}{3}\rho_{\perp}$ . However, for a system where the majority of the magnetic domains have their moments in the film plane, which is assumed to be the case in the Fe/V systems, the average resistivity can be defined as

$$\rho_{\text{av}} = \frac{1}{2}(\rho_{\parallel} + \rho_{\perp}). \quad (3)$$

Since the Fe/V samples exhibit very small hysteresis effects, the measured zero field resistivity  $\rho_0$  represents the resistivity of a randomly demagnetized system and is therefore approximately equal to  $\rho_{\text{av}}$ .<sup>17</sup> According to Eq. (3), the perpendicular resistivity then equals  $\rho_{\perp} = 2\rho_0 - \rho_{\parallel}$ . Inserting this expression in Eq. (2), the following equation for the anisotropic magnetoresistance for the Fe/V films is obtained:

$$\Delta\rho/\rho = 2(\rho_{\parallel} - \rho_0)/\rho_0. \quad (4)$$

In Fig. 5 the anisotropic magnetoresistance  $\Delta\rho/\rho$  (at 10 K), defined according to Eq. (4), is plotted vs the thickness of the V interlayers. The magnetic field was applied in the magnetically hard [110] direction. As can be seen, the magnitude of  $\Delta\rho/\rho$  decreases monotonically with increasing thickness of the V interlayers. A remarkable feature is the difference in sign in the anisotropic magnetoresistance between the pure Fe 500-Å film, which has a negative magnetoresistance  $\Delta\rho/\rho \approx -1.5\%$ , and the Fe/V (1 ML) with a positive value  $\Delta\rho/\rho \approx 7\%$ . A negative magnetoresistance in the parallel direction at low temperatures has also been found in iron single crystals.<sup>18</sup> In Fig. 6 the zero-field resistivity at 10 K,  $\rho_0(10 \text{ K})$ , and the residual resistivity ratio  $r_R = \rho_0(300 \text{ K})/\rho_0(10 \text{ K})$

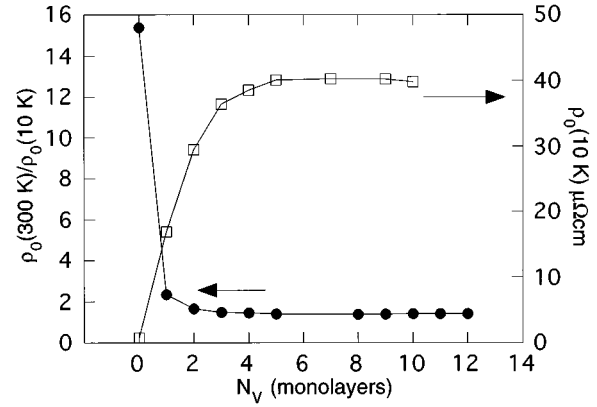


FIG. 6. Residual resistivity ratio  $\rho_0(300 \text{ K})/\rho_0(10 \text{ K})$  (circles) and the resistivity at 10 K,  $\rho_0(10 \text{ K})$  (squares) vs the number of vanadium layers.

$\rho_0(10 \text{ K})$  is plotted as a function of the number of atomic V layers. With increasing V thickness the  $r_R$  and  $\rho_0(10 \text{ K})$  values rapidly decrease and increase respectively for small thicknesses ( $0 < x < 3-4$  ML) and both saturate to constant values for V thicknesses  $3-4 < N_V < 12$  ML.

#### IV. DISCUSSION

In a multilayered system the resistivity is, in general, larger than the expected resistivity, assuming the system as consisting of independent parallel bulk resistors. According to Gurvitch,<sup>19</sup> the modulation length  $\lambda = d_1 + d_2$  (where  $d_1$  and  $d_2$  are the thicknesses of the different materials and  $d_1 \approx d_2$ ) plays an important role and, in some range of  $\lambda$ , the resistivity frequently follows a power law  $\rho \propto \lambda^{-1}$ . This occurs in a regime where the thicknesses of the layers limit the mean free path of the conduction electrons, due to scattering effects at the interfaces. A further decrease of  $\lambda$  enforces the resistivity to saturate at a constant value. It appears reasonable that for a superlattice with  $d_1 \gg d_2 \approx 1-3$  ML, the system can, from a conductivity point of view, be considered as an alloy, if the mean free path of the conduction electrons  $l$  is larger than the modulation length  $l > \lambda$ . In the Fe/V system,  $\rho_0(10 \text{ K})$  increases and  $r_R$  decreases with increasing number of vanadium layers for  $N_V < 3-4$  ML. In this range it is assumed that  $l > \lambda$  and that the system can be regarded as an iron-rich alloy with the approximate V concentration  $c_V = N_V/(N_V + N_{\text{Fe}})$ , where  $N_V$  and  $N_{\text{Fe}}$  are the number of atomic layers of the respective element. In Fig. 7 the anisotropic magnetoresistance is plotted vs  $c_V$ . Also included in the figure is anisotropic magnetoresistive data from a polycrystalline iron-vanadium alloy measured at 4.2 K by Sueda and Fujiwara.<sup>20</sup> With increasing V concentrations  $\Delta\rho/\rho$  for the alloy increases for small  $c_V$ , reaches a maximum at a concentration  $\approx 7\%$ , and decreases at larger  $c_V$ . An interesting fact is that for V concentrations larger than 7% the magnitude of  $\Delta\rho/\rho$  and the variation of  $\Delta\rho/\rho$  with respect to  $c_V$  is similar for the alloy and the superlattice. This result supports the assumption of an apparent alloying effect between Fe and V in the superlattice.

The anisotropic magnetoresistance originates, according to Smit,<sup>21</sup> from the spin-orbit coupling that imposes a mixing

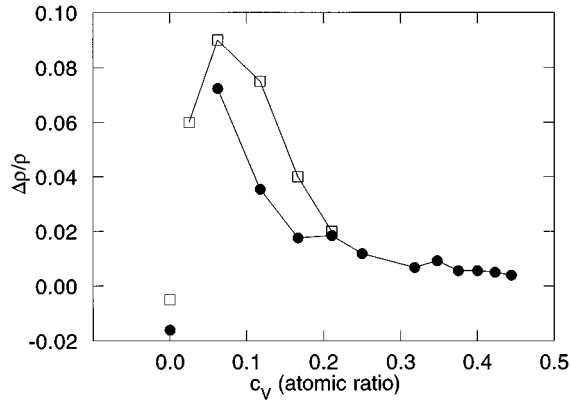


FIG. 7. Anisotropic magnetoresistance  $\Delta\rho/\rho$  for an Fe/V alloy at 4.2 K (squares) according to Sueda and Fujiwara (Ref. 20) and  $\Delta\rho/\rho$  at 10 K for the Fe/V superlattice (circles) plotted vs the V concentration  $c_V$ . The V concentration for the superlattice is defined as  $c_V = N_V / (N_V + N_{Fe})$ .

of spin-up and spin-down electron states in the  $d$ -electron bands. This results in a scattering mechanism (scattering of  $s$  electrons into empty  $d$  states at the Fermi level) that is strongly dependent on the relative orientation of the conduction electron  $k$  vector and the magnetization direction. In an extended theory, based on the model by Smit, Campbell *et al.*<sup>16,22</sup> proposed the following expression for the anisotropic magnetoresistance in Ni-based alloys:

$$\Delta\rho/\rho = (\alpha - 1)\gamma. \quad (5)$$

Here  $\alpha = \rho_{\downarrow}/\rho_{\uparrow}$  is the ratio between the resistivities for spin-down and spin-up electrons and  $\gamma$  is the spin-orbit coupling parameter. In Fe-based alloys, where  $\rho_{\uparrow} > \rho_{\downarrow}$ , Eq. (5) still holds if the ratio between the respective resistivity channels is defined as  $\alpha = \rho_{\uparrow}/\rho_{\downarrow}$ . In alloys with weak-scattering potentials, the amplitude of the  $3d$ -electron wave functions is only weakly affected by the impurity and hence the matrix element describing the scattering mechanism is assumed to be independent of the alloy composition.<sup>23</sup> This implies that  $\rho_{\downarrow}/\rho_{\uparrow}$  in Eq. (5) can be replaced by  $D_{d\downarrow}(\epsilon_F)/D_{d\uparrow}(\epsilon_F)$ , where  $D_d$  is the density of states of the  $d$  band. In the presence of a strong-scattering potential, as is the case for FeV alloys, the weak-scattering potential approximation  $\Delta\rho/\rho \approx D_{d\downarrow}(\epsilon_F)/D_{d\uparrow}(\epsilon_F)$  is not valid. In this case the anisotropic magnetoresistance can be calculated using the ‘‘split-band’’ model.<sup>24</sup> In this picture, a maximum in  $\rho_{\uparrow}$  vs V concentration for the FeV alloy is found, which is attributed to a

scattering resonance located in the spin-up  $3d$ -electron subband for Fe near the boundary between the Fe and V subbands. This yields a large value of  $\alpha = \rho_{\uparrow}/\rho_{\downarrow}$  and hence a large  $\Delta\rho/\rho$  according to Eq. (5). The results obtained using this model and the present experimental results support the assumption that the Fe/V films ( $N_V < 3-4$  ML) from a conductivity point of view behave as a solid solution of Fe and V; i.e., in the thin-film limit, the vanadium acts as impurity atoms imposing strong-scattering potentials in the iron lattice.

The predominance of the anisotropic magnetoresistance over a possible giant magnetoresistance effect in the single-crystalline Fe/V ( $1 < N_V < 12$  ML) multilayer system may be an effect of the absence of an antiferromagnetic coupling. The reported experimental results of an oscillating magnetic coupling<sup>3</sup> and a spin valve related magnetoresistance<sup>11</sup> in Fe/V multilayers have been obtained using polycrystalline samples with an out-of-plane texture different from the [001] growth direction. According to calculations using a first-principle Green’s-function technique, an oscillating magnetic coupling does exist in a bcc Fe/V(001) trilayer system with ideal interfaces.<sup>10</sup> This result is consistent with a possible nesting  $k$  vector extracted from the Fermi surface of vanadium in the [001] direction.<sup>10</sup> To account for a real system with roughness at the interfaces an average coupling strength was modeled as

$$J_{av}(N_V) = \frac{1}{4}[J(N_V - 1) + 2J(N_V) + J(N_V + 1)]. \quad (6)$$

This effective magnetic coupling is positive and rapidly decays with increasing thickness of V and becomes insignificant for  $N_V > 6-7$  ML. The absence of an oscillating magnetic coupling in the present measurements might be attributed to a finite but small roughness at the interface, which obscures the oscillatory behavior. To obtain a better understanding of the magnetic properties of this system careful investigations on Fe/V superlattices grown in different directions are necessary. Investigations on Fe/V(011) are in progress and results are planned to be presented in a forthcoming paper.

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